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# ELECTRODEPOSITION OF EROSION AND OXIDATION RESISTANT COATINGS FOR GRAPHITE

FINAL SUMMARY REPORT

15 March 1963

VECO

VALUE ENGINEERING COMPANY

2320 Jefferson Davis Highway • Alexandria, Va. • King 8-8300

Final Summary Report

# Electrodeposition of Erosion and Oxidation Resistant Coatings for Graphite

Conducted for:

Navy Department
Bureau of Naval Weapons

Contract No. N600(19)58317

15 March 1963

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## ABSTRACT

Research was conducted to optimize electrodeposited metal-ceramic composite coatings for protection of graphite rocket nozzles against oxidation and erosion. A series of nozzles were coated with different thicknesses of the cermet and fired in the company's gaseous hydrogen-gaseous oxygen rocket motor. The data from these firings indicated that 2.0 to 2.5 is the optimum coating thickness. The effect of the concentration of ceramic particles in the plating bath on the amount of ceramic occluded in the coating was investigated. Results of this study showed that there was no effect on the amount of particles in the coating.

A series of ATJ nozzles were coated, given different heat treatments and fired in a solid propellant motor. Although there was no significant improvement over uncoated graphite for the 60 second firings, a number of conclusions could be drawn from the firing data. Nozzles which were heated in argon before heat treatment were superior to nozzles without the argon treatment. In addition, the chromium-zirconium diboride + 10% molybdenum disilicide cermet was better than the chromium-silicon nitride cermet tested. A nozzle coated from a bath containing a proprietary dispersing agent exhibited a superior performance.

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## INTRODUCTION

This report summarizes the work performed by Value Engineering Company during the ten-month duration of the contract. The contract period was 17 April 1962 through 16 February 1963. Since a third quarterly report was not required, this report will cover the final four-month effort in detail.

The project was conducted under the technical direction of Mr. S. J. Matesky, Bureau of Naval Weapons, Code RMMP-23.

## BACKGROUND

During the period February 1961 through February 1962, Value Engineering Company conducted research for BuWeps on an electrodeposited metal-ceramic coating for protection of graphite. This work was performed under Contract NOw 61-0670-C. A final summary report was submitted on 22 March 1962.

The process as developed by Value Engineering Company utilizes conventional electroplating solutions with ceramic powders suspended in the solution. The particles of ceramic are thought to be charged and driven toward the cathode when sufficiently high current is applied. The process is in some respects similar to electrophoresis. However, in the Value Engineering Company process, a conductive solution is used whereas true electrophoresis utilizes an essentially non-conductive solution.

The work performed during 1961 consisted primarily of electrodeposition experiments with a variety of ceramics in combination with chromium metal. Of the twelve ceramics utilized, the following appeared to have the most potential. (Denoted by asterisk.)

## Ceramics

1. Hafnium Boride
* 2. Zirconium Boride
* 3. Zirconium Boride plus 10% Molybdenum Disilicide
4. Tungsten Boride
5. Tantalum Boride
6. Hafnium Carbide
* 7. Tantalum Carbide
8. Columbium Carbide
* 9. Silicon Carbide
10. Zirconium Oxide
11. Boron Nitride
* 12. Silicon Nitride

The earlier testing of the ceramic-metal coating consisted of oxyacetylene flame tests. Later in the program nozzles were tested in a hydrogen-oxygen rocket motor designed and constructed by Value Engineering Company.

The results of the experimental coating and evaluation program completed in February 1962 demonstrated the feasibility of electrodepositing a variety of metal-ceramic mixtures.

The program covered by this summary report was completed in February 1963. The prime objective was to optimize the electrodeposited metal-ceramic composition and the process to a point where it would be of use in protecting graphite rocket nozzles of current and future Navy missiles.

Considerable emphasis was placed on the aspects of process control and reproducibility of the coating.

## WORK PERFORMED

### Optimization of Coating Thickness

In order to determine the optimum coating thickness, eighteen ATJ graphite nozzles were coated with a chromium-zirconium oxide cermet coating. These nozzles had coating thicknesses ranging from 1 to 6 mils and were all heat treated at 1600°F for 5 minutes subsequent to coating. Testing was conducted in the company's hydrogen-oxygen rocket motor at a chamber pressure of 130-140 psi. The majority of these nozzles failed after 5 to 7 seconds with no significant difference between nozzles of different thicknesses. It is believed that these short failure times are probably due to poor adherence of the coating to the substrate caused by unfavorable heat treating conditions. Some of these nozzles were red, blue or purple after heat treatment which corresponds to different hydrates of chromium oxide. Nozzles which had lasted for long firing times were a green color after heat treatment. This green oxide is the unhydrated $Cr_2O_3$. It was, therefore, decided to coat an additional 18 nozzles in the 1 to 6 mil thickness range similar to those described above. These nozzles were then heat treated in a moisture free furnace atmosphere. All of these nozzles were converted to the green $Cr_2O_3$.

The nozzles were tested in the $H_2$-$O_2$ rocket motor. Coating thicknesses and the corresponding time to failure are tabulated in Table I. The data are plotted in Figure 1. The uncoated ATJ graphite nozzles used as a standard, failed after approximately 7 to 9 seconds. As can be seen from Figure 1, coatings 4 to 6 mils thick are unsatisfactory. The optimum coating thickness appears to be 2 to 2.5 mils. Figures 2 and 3 illustrate sectioned nozzles.

| NOZZLE NUMBER | THICKNESS (mils) | TIME TO FAILURE (sec) |
|---|---|---|
| 135 | 1.0 | 17 |
| 144 | 1.5 | 13.75 |
| 129 | 1.5 | 13 |
| 142 | 2.0 | 13 |
| 65 | 2.5 | 14 |
| 31 | 2.5 | 14.5 |
| 124 | 3.0 | 11.75 |
| 150 | 3.0 | 15.5 |
| 130 | 3.5 | 10.5 |
| 126 | 4.0 | 11 |
| 133 | 4.5 | 9 |
| 160 | 4.5 | 7 |
| 131 | 4.5 | 8.5 |
| 168 | 5.0 | 5 |
| 153 | 5.0 | 9 |
| 159 | 5.5 | 6 |
| 151 | 6.0 | 8.5 |
| 134 | 6.5 | 5 |

Table 1: Effect of Coating Thickness on Life of Test Nozzles

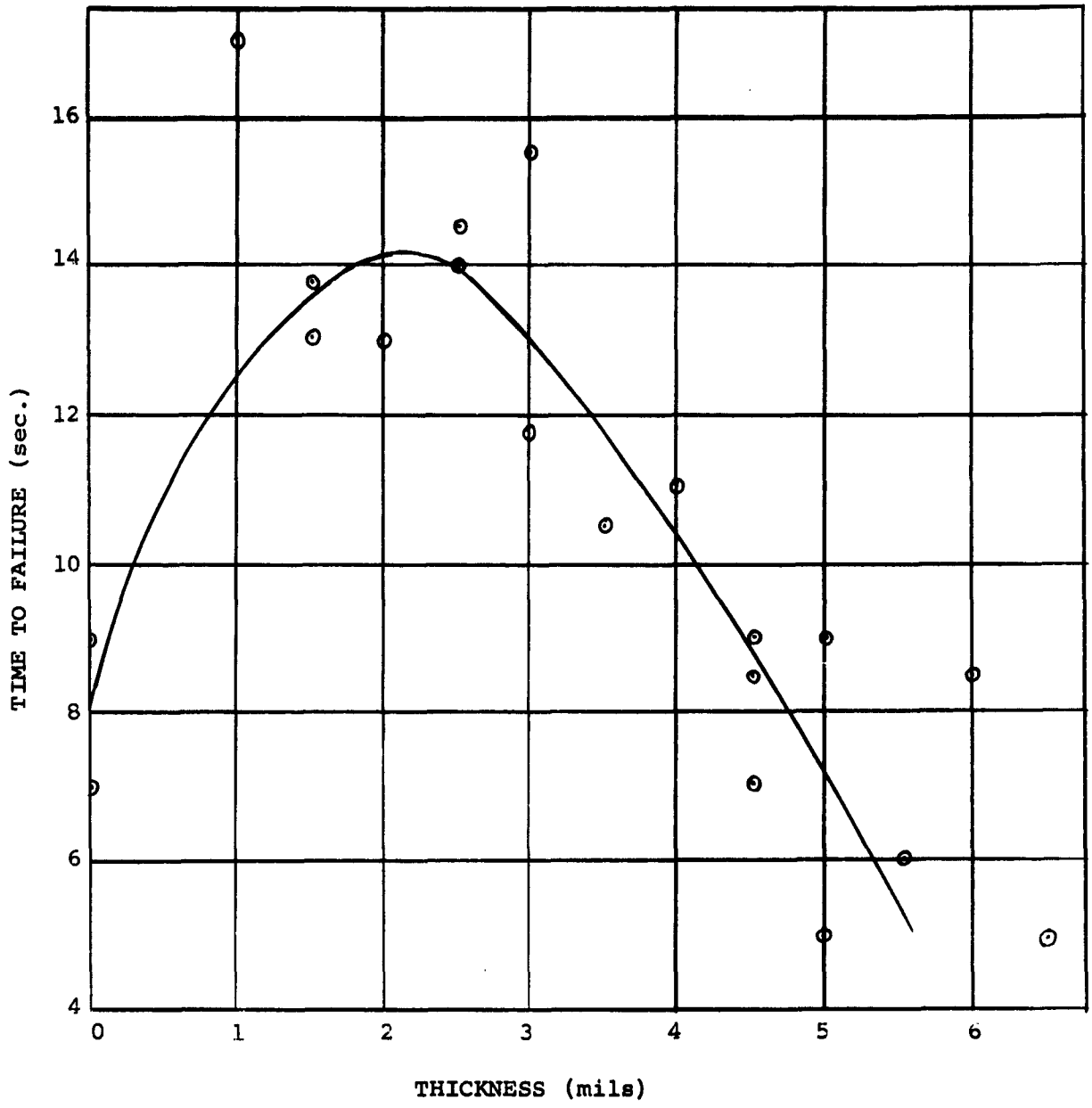



FIGURE 1: Time to Failure vs. Coating Thickness for Chromium-Zirconium Oxide Coated Graphite Nozzles

Figure 2. Sectioned VECO Test Nozzle Coated With Cr-$ZrO_2$ Cermet. Nozzle Was Heated to 1600°F Subsequent to Application of Coating. Unfired Nozzle, Actual Size.

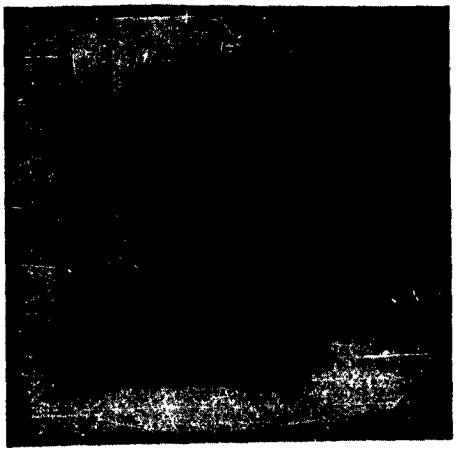


Figure 3. Sectioned VECO Test Nozzle Coated And Heat Treated As Above After Firing for 18 Seconds in $H_2$ - $O_2$ Test Motor.

## Effect of Particle Concentration

An attempt was made to determine whether the amount of ceramic particles in the plating bath was related to the amount of particles in the coating. Five baths with increasing amounts of ceramic particles were made up. Two graphite blocks were plated from each one of these baths. These blocks were then sectioned and studied metallographically to determine the volume of ceramic particles in the coating.

Five baths having increasing amounts of ceramic particles were prepared with the following quantities:

a. 60 ml $ZrO_2$ - 7500 ml solution

b. 120 ml $ZrO_2$ - 7500 ml solution

c. 200 ml $ZrO_2$ - 7500 ml solution

d. 300 ml $ZrO_2$ - 7500 ml solution

e. 400 ml $ZrO_2$ - 7500 ml solution

The effect of particle concentration in these solutions was to be studied by metallographic examination of coated graphite blocks and by $H_2$ - $O_2$ rocket motor testing of coated graphite nozzles.

Two ATJ graphite blocks and two ATJ test nozzles were coated from each of the baths. The graphite blocks were sectioned, mounted, and polished for metallographic study to determine the quantity, uniformity, and other characteristics of the ceramic particles.

Very little was determined from the metallographic examination. The blocks coated from the solutions having greater concentrations appeared to have no increased concentration of particles in the coating.

This experiment was repeated utilizing silicon carbide powder to determine if another ceramic would act differently. A variation from 100 to 300 ml of SiC resulted in no significant differences in the coatings examined microscopically.

These experiments proved that merely "overloading" the solution with ceramic is not the answer to obtaining more ceramic particles in the coating.

Effect of Coating Composition

Work performed under a previous contract indicated that zirconium boride-10% molybdenum disilicide, silicon nitride, tantalum carbide or silicon carbide in a chromium matrix were superior to the other cermet systems studies.

In this program experiments were conducted to investigate the effect of coating composition utilizing what was determined to be an optimum coating thickness of 2 1/2 to 3 mils. Evaluation was conducted on ATJ test nozzles similar to those previously used in the program. Plating solutions were prepared for depositing each of the above mentioned ceramics in a chromium matrix. Six nozzles were coated from each of these solutions.

The twenty-four nozzles were fired in Value Engineering Company's $H_2$ - $O_2$ test motor. Chamber pressure during the tests was in the range of 130-150 psi. The results were inconclusive as each of the nozzles showed rapid erosion after eight seconds of firing time.

Since preliminary tests conducted earlier in the program indicated these ceramics to be superior, it is believed that factors related to the $H_2$ - $O_2$ motor itself resulted in failure of all of the nozzles.

Visual examination of the nozzles after firing revealed that the coating had failed as a result of thermal shock, rather than by straight erosion or melting.

Nozzle Evaluation

The final phase of this program involved an evaluation of a number of coated nozzles in a solid propellant motor at the facilities of Atlantic Research Corporation.

The propellant utilized was Arcite 373 which has a theoretical flame temperature of 5600°F. Chamber pressure was to be in the 900 to 1000 psi range.

The 373 propellant is an O-max type in which there is just the necessary amount of oxidizer to react with the fuel. The erosion rate for uncoated ATJ under these conditions has been approximately 1 mil per second.

A series of nozzles were coated and post treated under different conditions as shown in the table below.

| Ceramic | No. of Nozzles | Coating Post Treatment |
|---|---|---|
| $Si_3N_4$ | 2 | 1600°F for 5 min. |
| | 2 | 2200°F for 1 hr in Argon |
| | 2 | 2200°F for 1 hr in Argon and cooled in air |

| Ceramic | No. of Nozzles | Coating Post Treatment |
|---|---|---|
| $ZrB_2$-$MoSi_2$ | 2 | 1600°F for 5 min. |
| | 2 | 2200°F for 1 hr in Argon |
| | 2 | 2200°F for 1 hr in Argon and cooled in air |

The purpose of the heat treatment at 1600°F for 5 minutes was to convert the surface chromium of the cermet to chromium oxide, $Cr_2O_3$. This has the advantage of having $Cr_2O_3$ with its 4109°F melting point on the surface as opposed to chromium with its melting point of 3434°F.

The purpose of the 2200°F treatment in argon was to cause some diffusion of the cermet coating into the graphite substrate thus increasing the adhesion.

The nozzles heated in argon at 2200°F for 1 hour were allowed to cool in air. It was anticipated that the treatment in argon would cause diffusion and improve the adhesion and the cooling in air would cause oxidation of the surface chromium to chromium oxide.

Previous studies had shown that graphite should have an 80 RMS finish in order to obtain maximum adhesion and performance in a rocket nozzle. Firings with coatings of different thicknesses showed that the optimum thickness is 2 to 3

mils, Consequently, all nozzles prepared for firing in the solid propellant motor had an 80 RMS finish and 2 to 3 mils of coating.

Results of Solid Propellant Firings

The results of the firing tests are recorded in the pressure-time curves, Figures 4 through 16 and are summarized in Table II.

The average erosion rates of the coated nozzles varied from 0.60 to 0.89 mil/sec. There was only one uncoated ATJ nozzle fired for comparison. This nozzle eroded at a lower rate of 0.56 mil/sec.

It is apparent that the coating loses its adherence at some stage of the firing, however, the pressure-time curves do not clearly indicate the time at which the coating affords some protection to the graphite before its removal. In a 60-second firing test, the erosion rate may be high because of the fact that the largest erosion occurs during the last 30 to 40 seconds, when there is no longer any coating to protect the graphite at the throat. Perhaps a better evaluation of the effectiveness of the cermet coating would be a 20 or 30 second firing.

It is recognized that once the coating fails catastrophically, the bits of loose coating do more damage to

TABLE II

TEST MOTOR FIRINGS IN MATERIALS EVALUATION

| Round | Total[a] Burning Time ($t_a$) (seconds) | Maximum Pressure (psi) | Average[a] Pressure ($P_a$) (psi) | Ceramic | Heat[d] Treatment | Diameter of Throat Before (in) | Diameter of Throat After[b] (in) | Erosion Rate (mils/sec) |
|---|---|---|---|---|---|---|---|---|
| M-316 | 58.5 | 1022 | 754 | $Si_3N_4$ | 1 | 0.517 | 0.596<br>0.627 | 0.81 |
| M-317 | 55.1 | 995 | 742 | $ZrB_2$+$MoSi_2$ | 1 | 0.523 | 0.591<br>0.634 | 0.81 |
| M-318 | 60.5 | 1000 | 735 | $Si_3N_4$ | 1 | 0.531 | 0.620<br>0.656 | 0.89 |
| M-319 | 61.9 | 965 | 710 | $ZrB_2$+$MoSi_2$ | 1 | 0.526 | 0.604<br>0.628 | 0.73 |
| M-320 | 62.5 | 965 | 724 | $Si_3N_4$ | 2 | 0.528 | 0.605<br>0.624 | 0.70 |
| M-321 | 60.8 | 1000 | 733 | $Si_3N_4$ | 3 | 0.528 | 0.607<br>0.643 | 0.80 |
| M-322 | 63.7 | 964 | 715 | $Si_3N_4$ | 3 | 0.518 | 0.595<br>0.618 | 0.70 |
| M-323 | 61.4 | 940 | 703 | $Si_3N_4$ | 2 | 0.524 | 0.619<br>0.641 | 0.88 |
| M-324 | 61.3 | 989 | 714 | $ZrB_2$+$MoSi_2$ | 2 | 0.534 | 0.613<br>0.616 | 0.66 |

a. Total burning time and average pressure taken with respect to 50 per cent tail off.
b. Minimum and maximum throat dimensions.
c. All coatings 2-3 mils in thickness.
d. 1. 1600°F 5 minutes in air. 2. 2200°F 1 hour in argon + 1600°F 5 minutes in air.
3. 2200°F 1 hour in argon.

| Round | Total[a] Burning Time ($t_a$) (seconds) | Maximum Pressure (psi) | Average[a] Pressure ($P_a$) (psi) | Ceramic | Heat[d] Treatment | Diameter of Throat Before (in) | Diameter of Throat After[b] (in) | Erosion Rate (mils/sec) |
|---|---|---|---|---|---|---|---|---|
| M-325 | 61.8 | 990 | 729 | $ZrB_2$+$MoSi_2$ | 2 | 0.531 | 0.606<br>0.623 | 0.68 |
| M-326 | 62.3 | 968 | 728 | $ZrB_2$+$MoSi_2$ | 3 | 0.529 | 0.598<br>0.622 | 0.65 |
| M-331 | 63.0 | 962 | 732 | $ZrB_2$+ Dispersant | 1 | 0.528 | 0.595<br>0.613 | 0.60 |
| M-330 | 61.8 | 1000 | 774 | Uncoated ATJ | - | 0.529 | 0.587<br>0.609 | 0.56 |

the throat than no coating at all. This accounts for the lower erosion rate of the uncoated graphite nozzle.

Table III shows the erosion rates for each ceramic for each type of heat treatment.

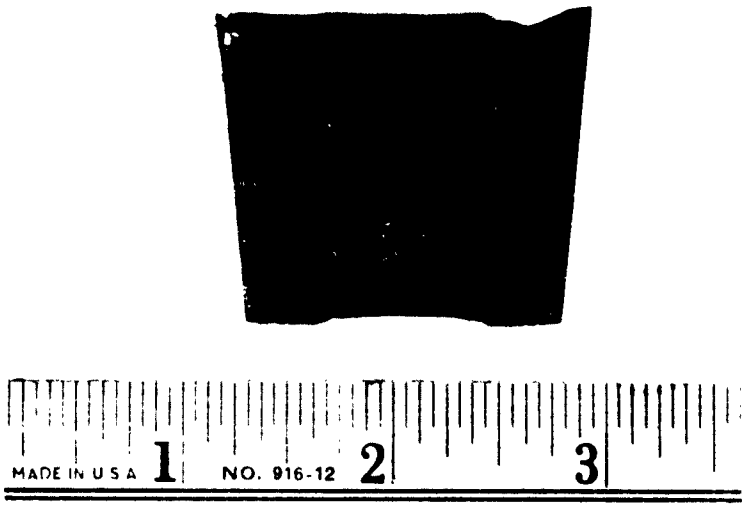
TABLE III

Erosion Rate (mils/sec)

| Heat Treatment | Zirconium Diboride + Proprietary Dispersant | Silicon Nitride | Zirconium Diboride + 10% Molybdenum Disilicide |
|---|---|---|---|
| 1600°F for 5 minutes | 0.60 | 0.89<br>0.81 | 0.81<br>0.73 |
| 2200°F for 1 hour in Argon | | 0.80<br>0.70 | 0.65 |
| 2200°F for 1 hour in Argon + 1600°F for 5 minutes | | 0.88<br>0.70 | 0.68<br>0.66 |

Several conclusions can be drawn from these data. Nozzles coated from baths containing the zirconium diboride-molybdenum disilicide mixture performed better than nozzles coated with silicon nitride. In addition, nozzles which were given the argon treatment were superior to those which were not heat treated in this manner.

One nozzle was coated from a bath containing zirconium diboride and a proprietary dispersing agent and heat treated at 1600°F for 5 minutes. The erosion rate of .60 mils/sec was considerably lower than the 0.89 and 0.81 mils/sec for $Si_3N_4$ and the 0.81 and 0.73 mils/sec for $ZrB_2$-$MoSi_2$ heat treated at the same conditions. If the nozzle coated in the bath with the dispersant were given the argon treatment it would have conceivably performed even better.




Fired ARC Nozzle

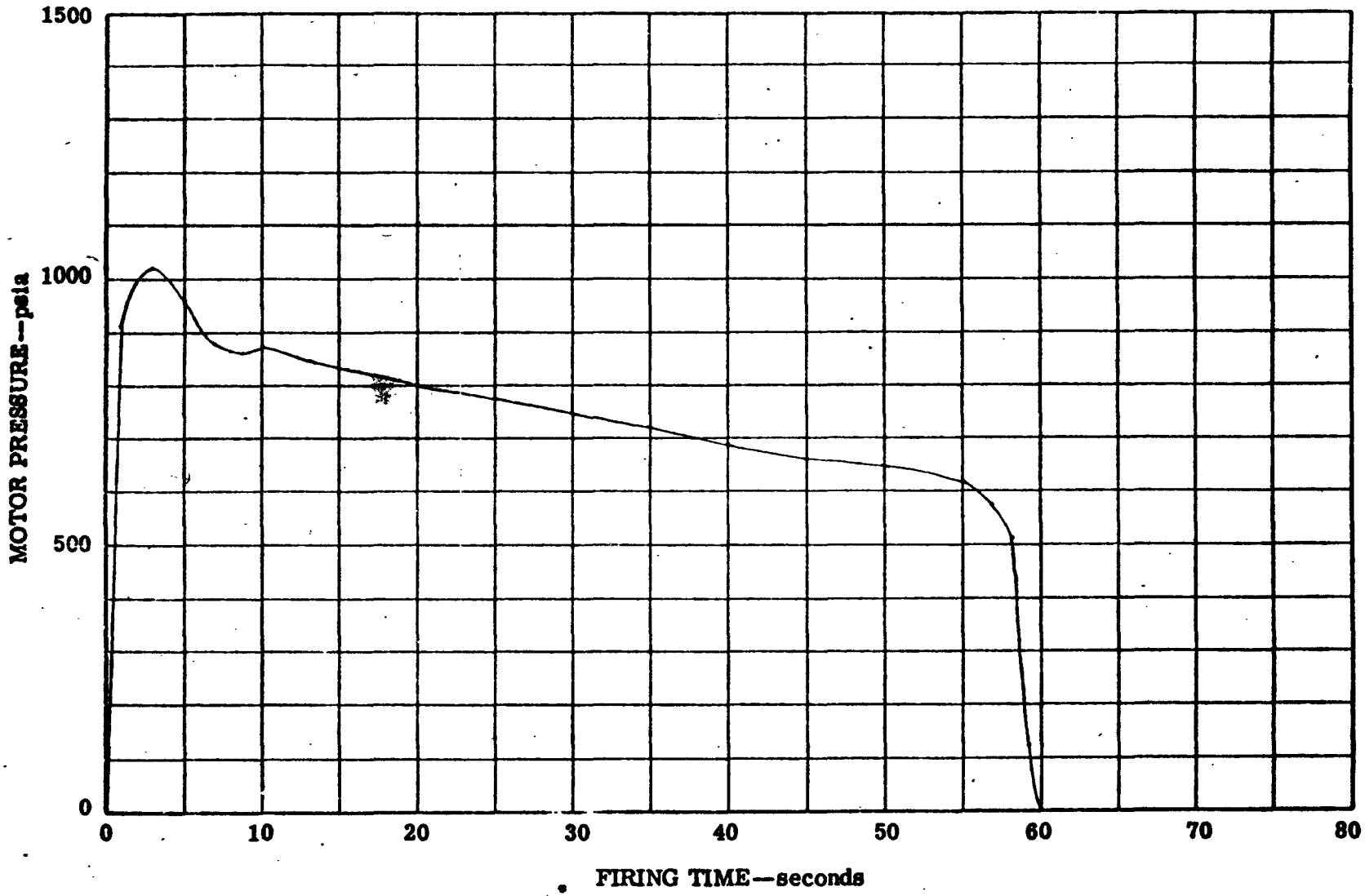



Figure 4. Motor Pressure Trace for Firing M-316

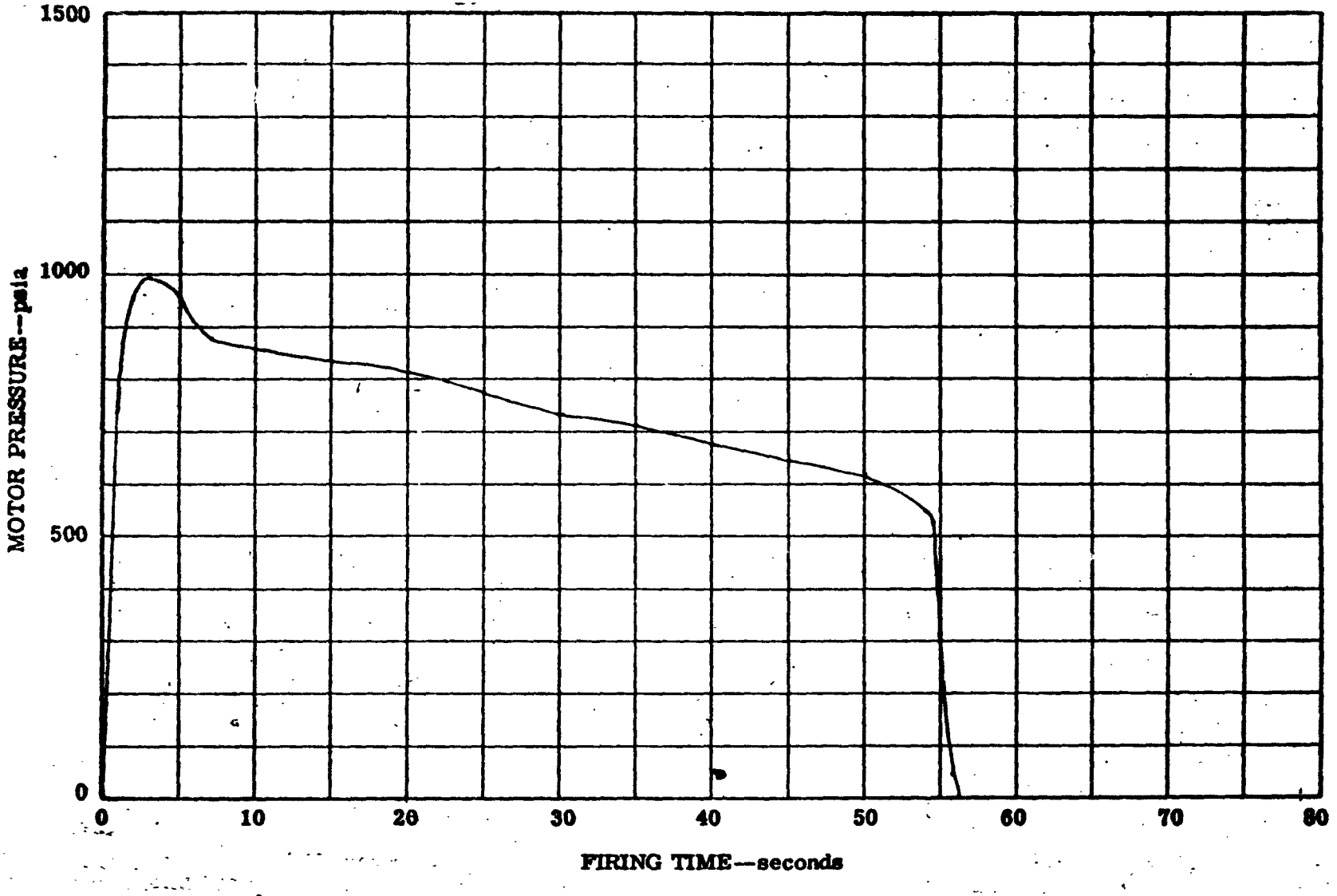



Figure 5. Motor Pressure Trace for Firing M-317

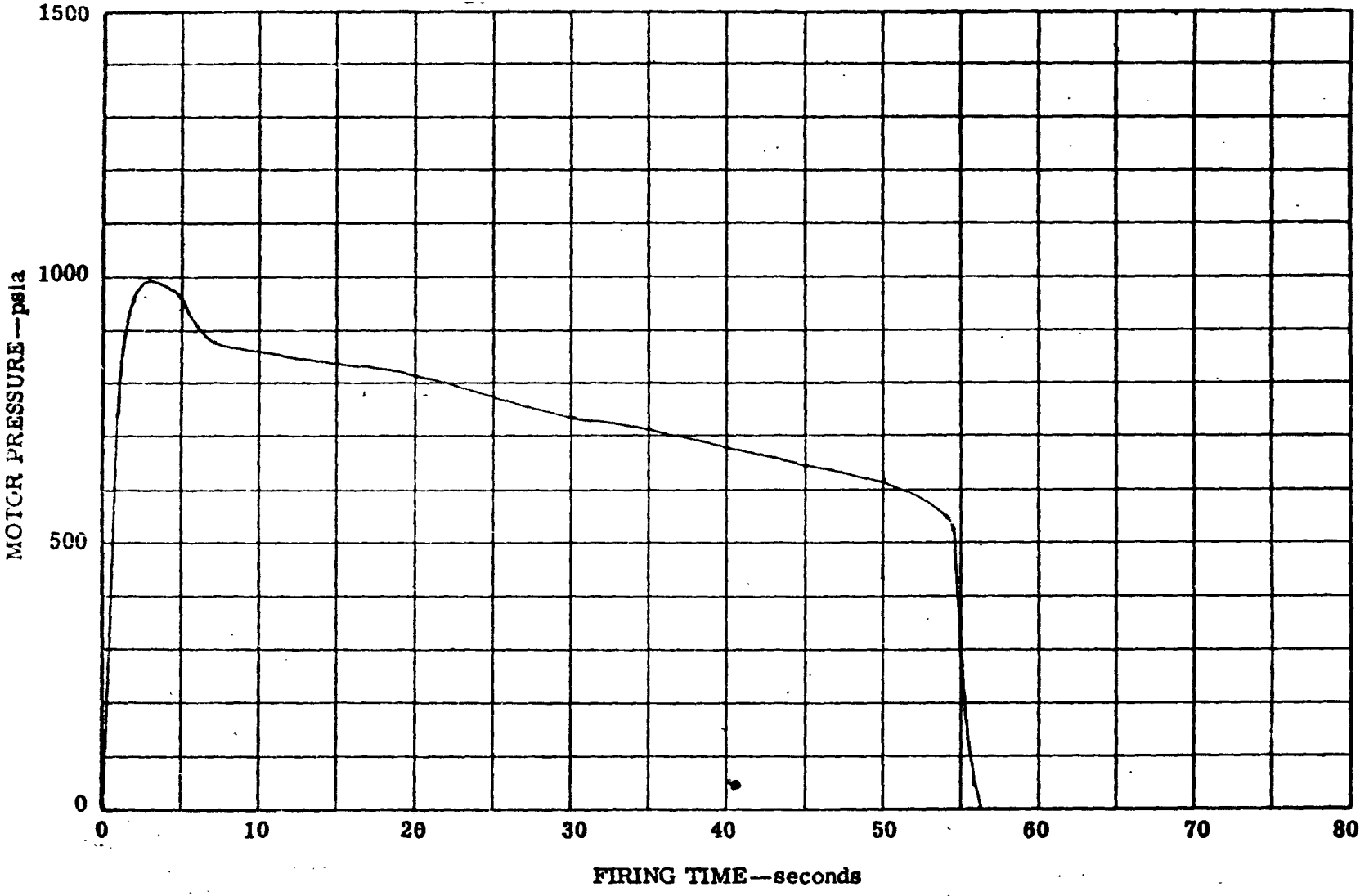



Figure 5. Motor Pressure Trace for Firing M-317

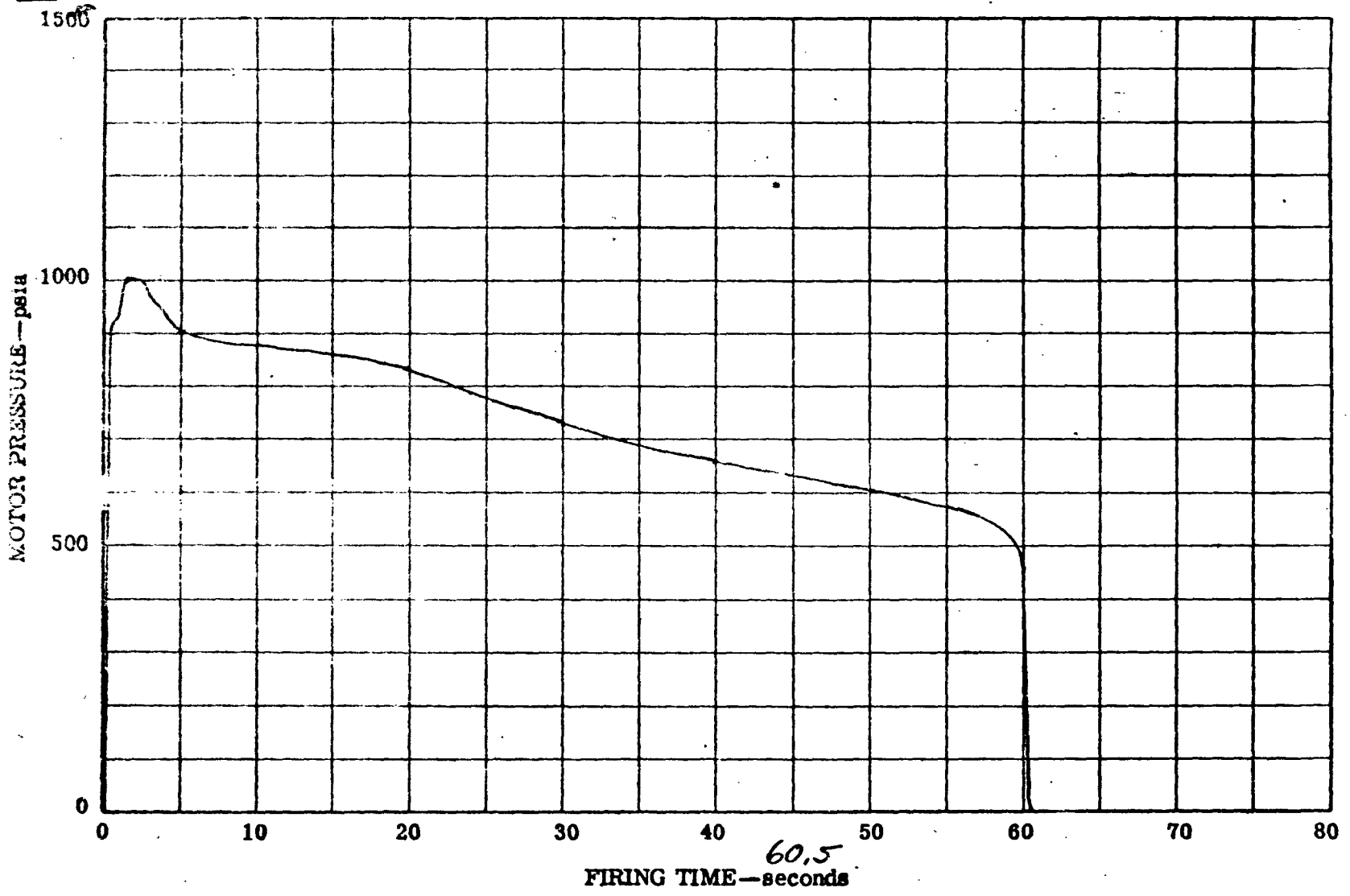



Figure 6 . Motor Pressure Trace for Firing M-318

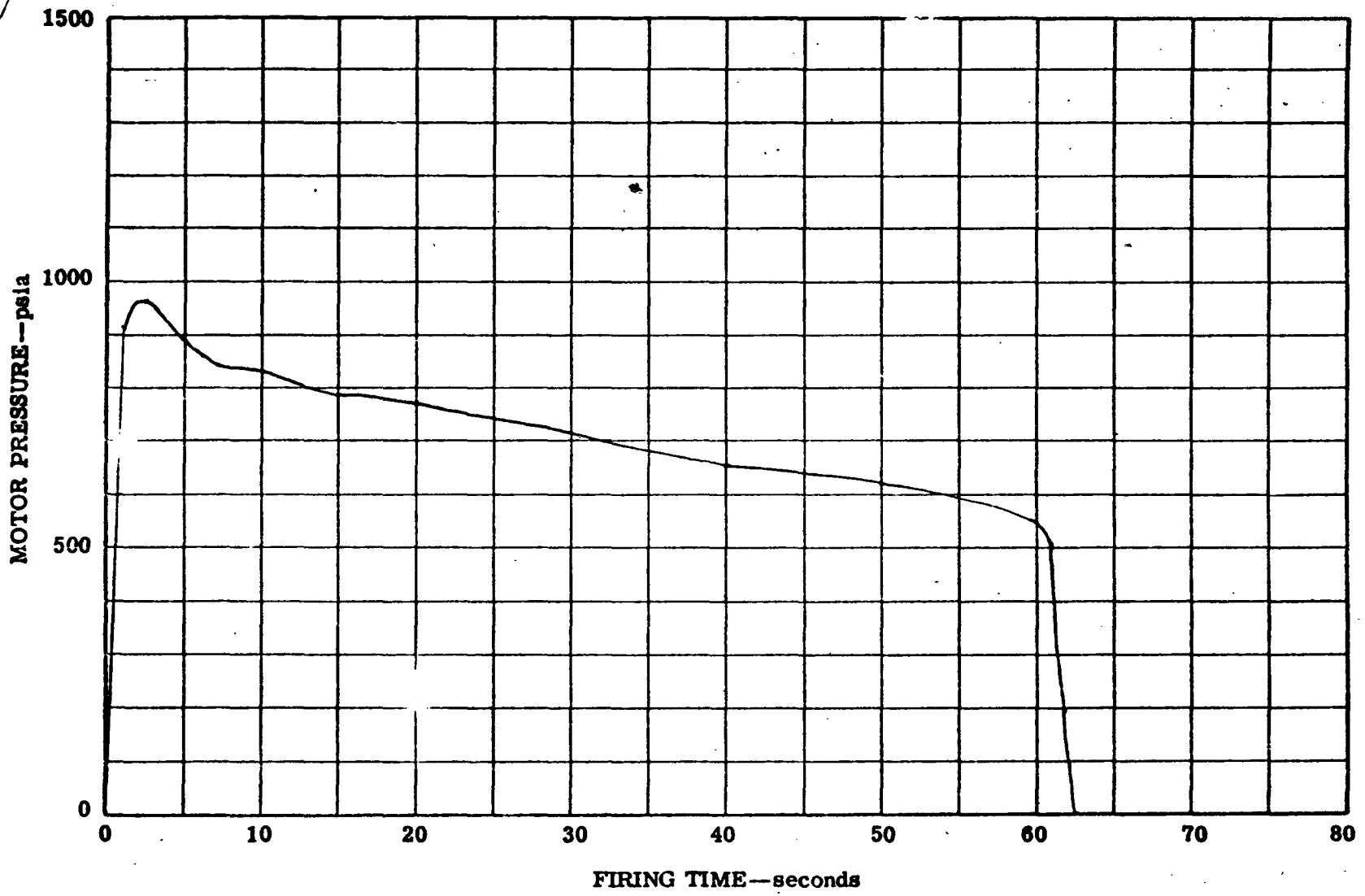



Figure 7. Motor Pressure Trace for Firing M-319

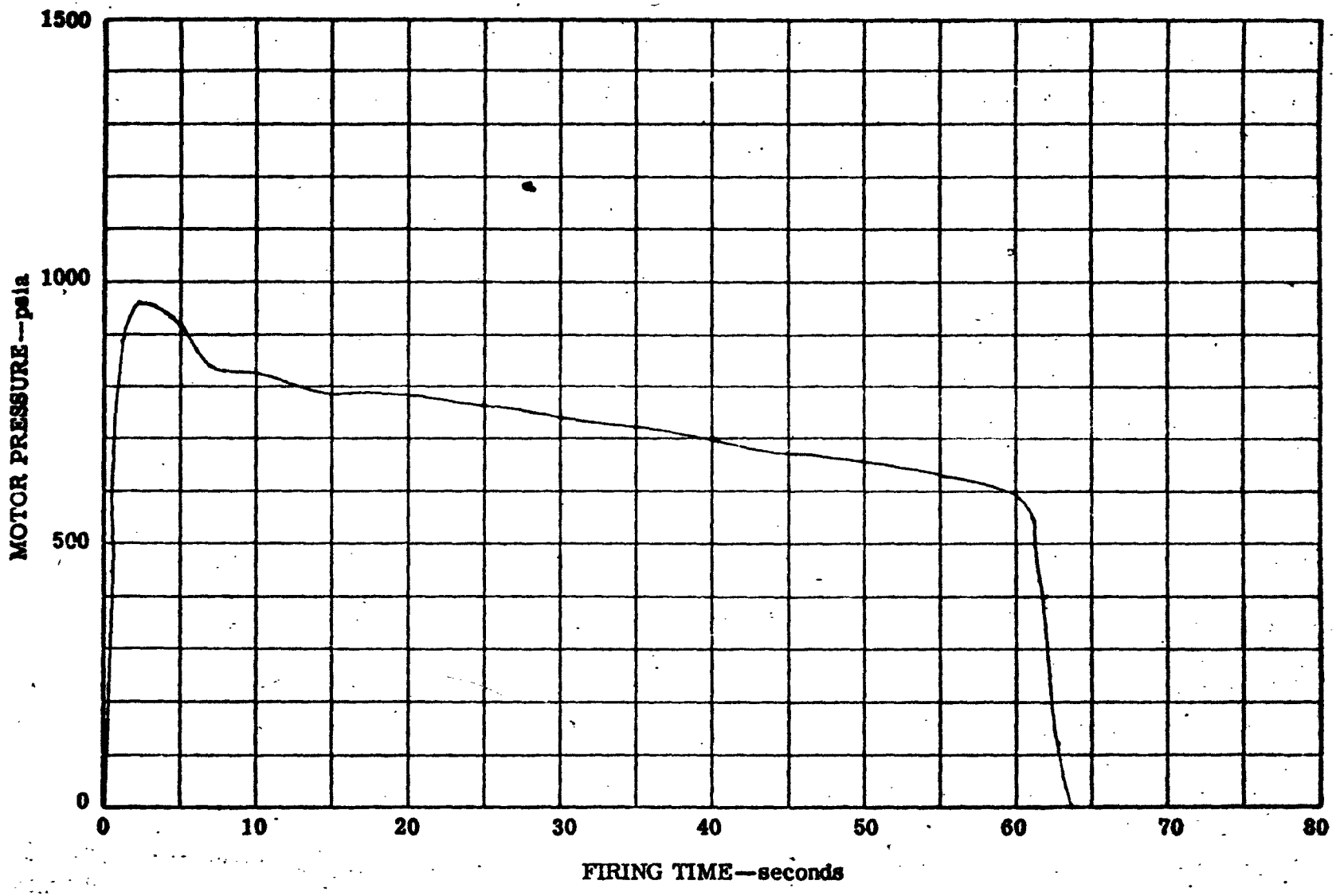



Figure 8. Motor Pressure Trace for Firing M-320

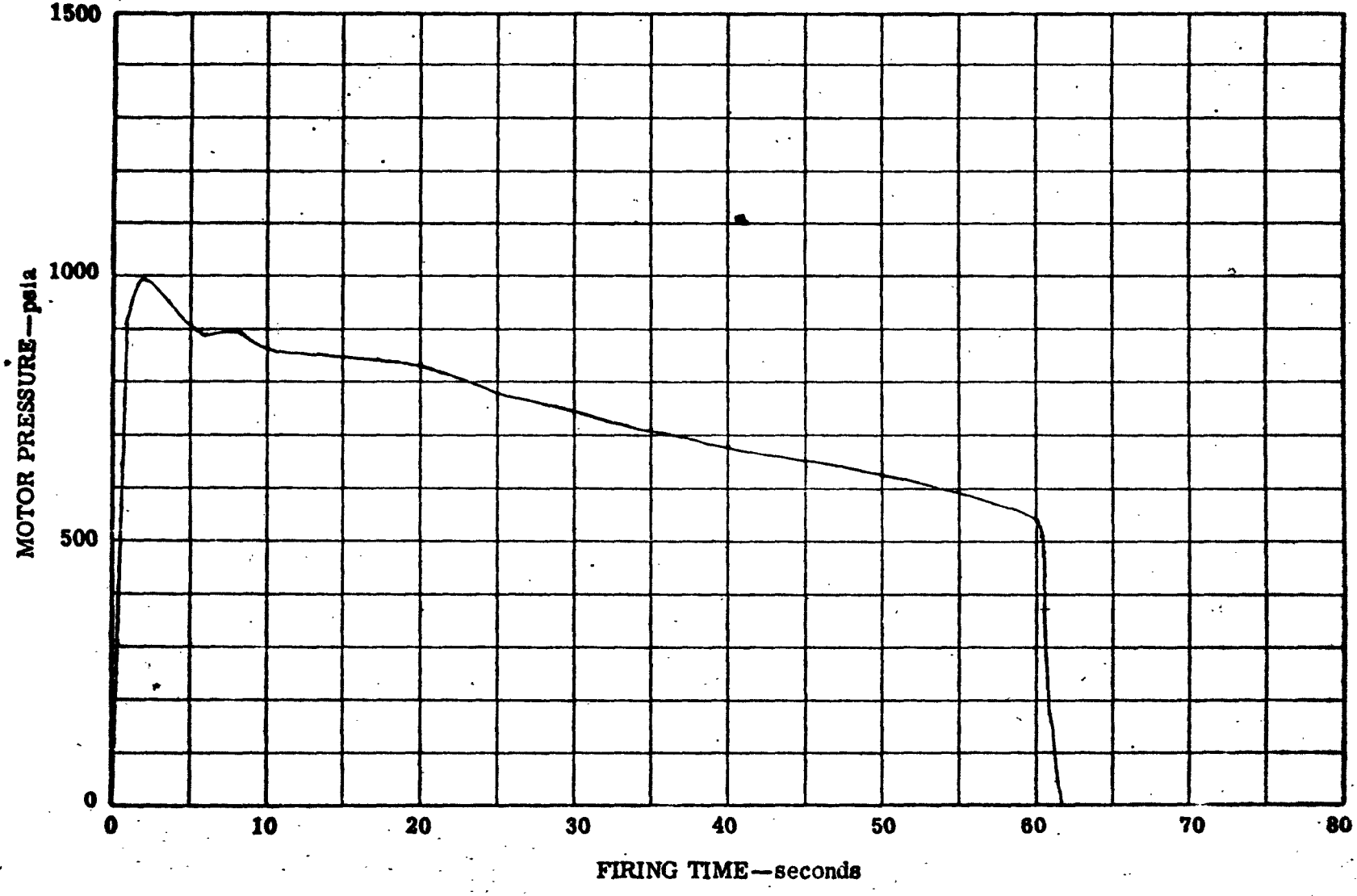



Figure 9. Motor Pressure Trace for Firing M-32 J

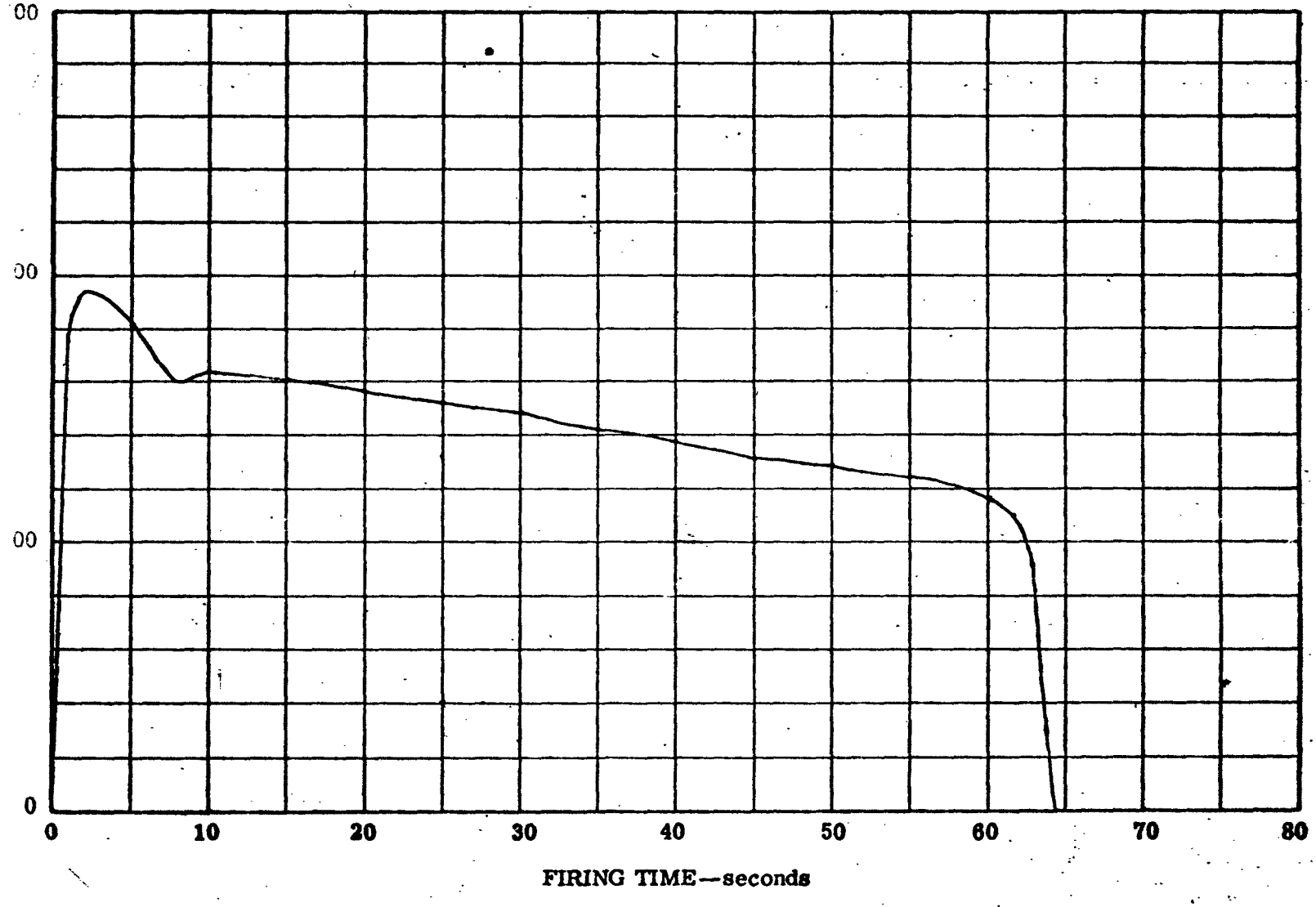



Figure 10. Motor Pressure Trace for Firing M-322

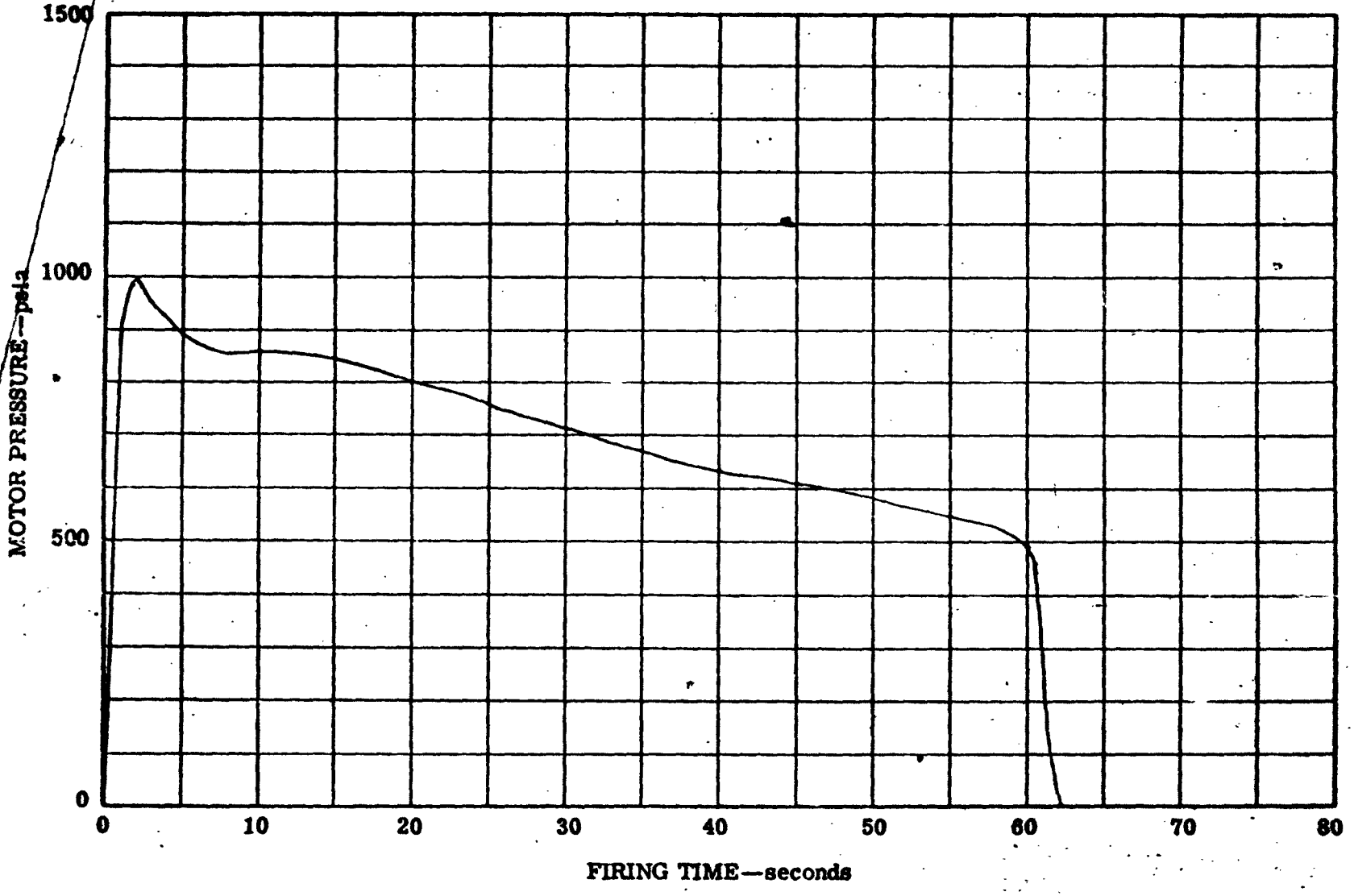



Figure 11 . Motor Pressure Trace for Firing M-323

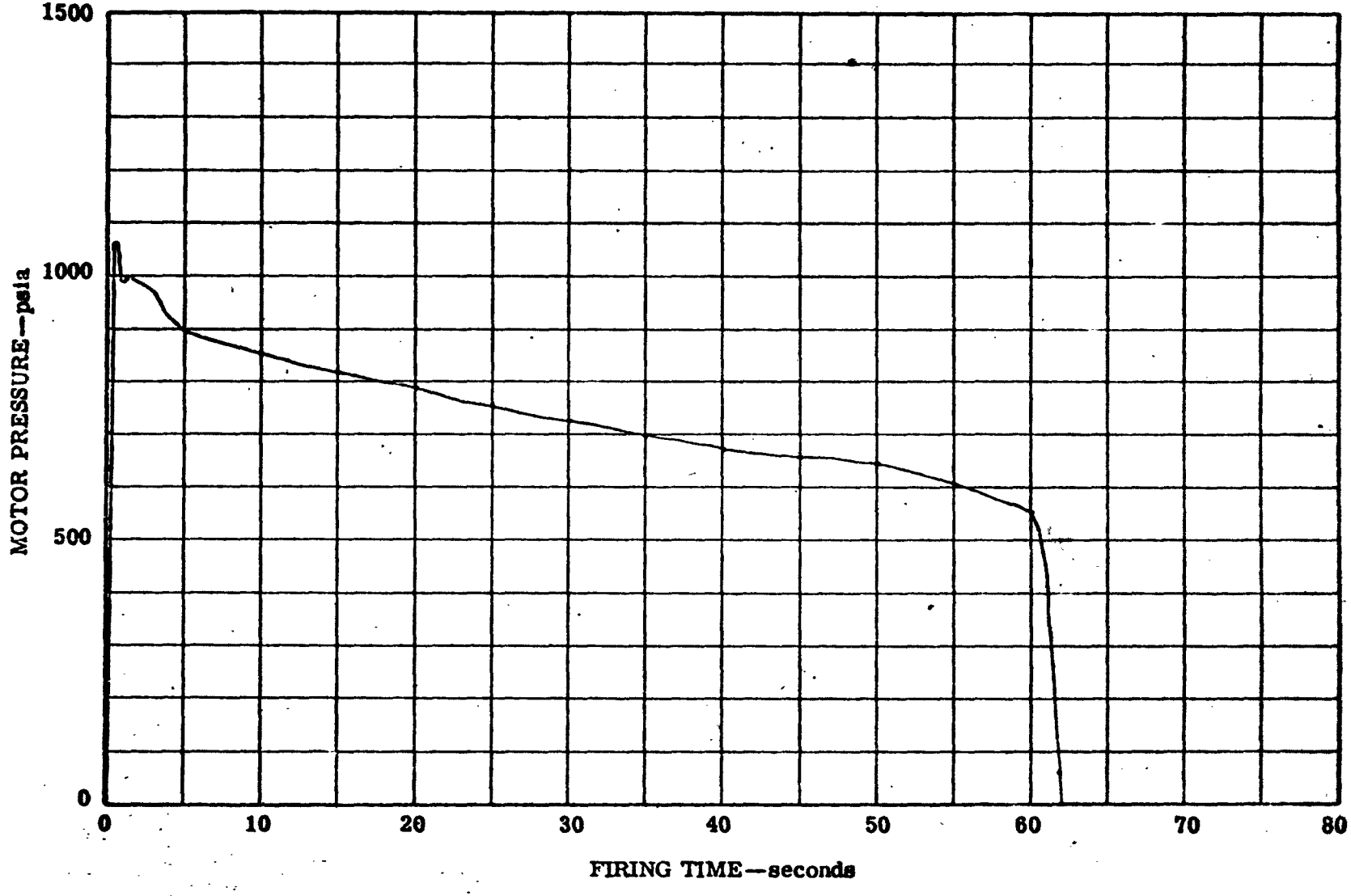



Figure 12. Motor Pressure Trace for Firing M-324

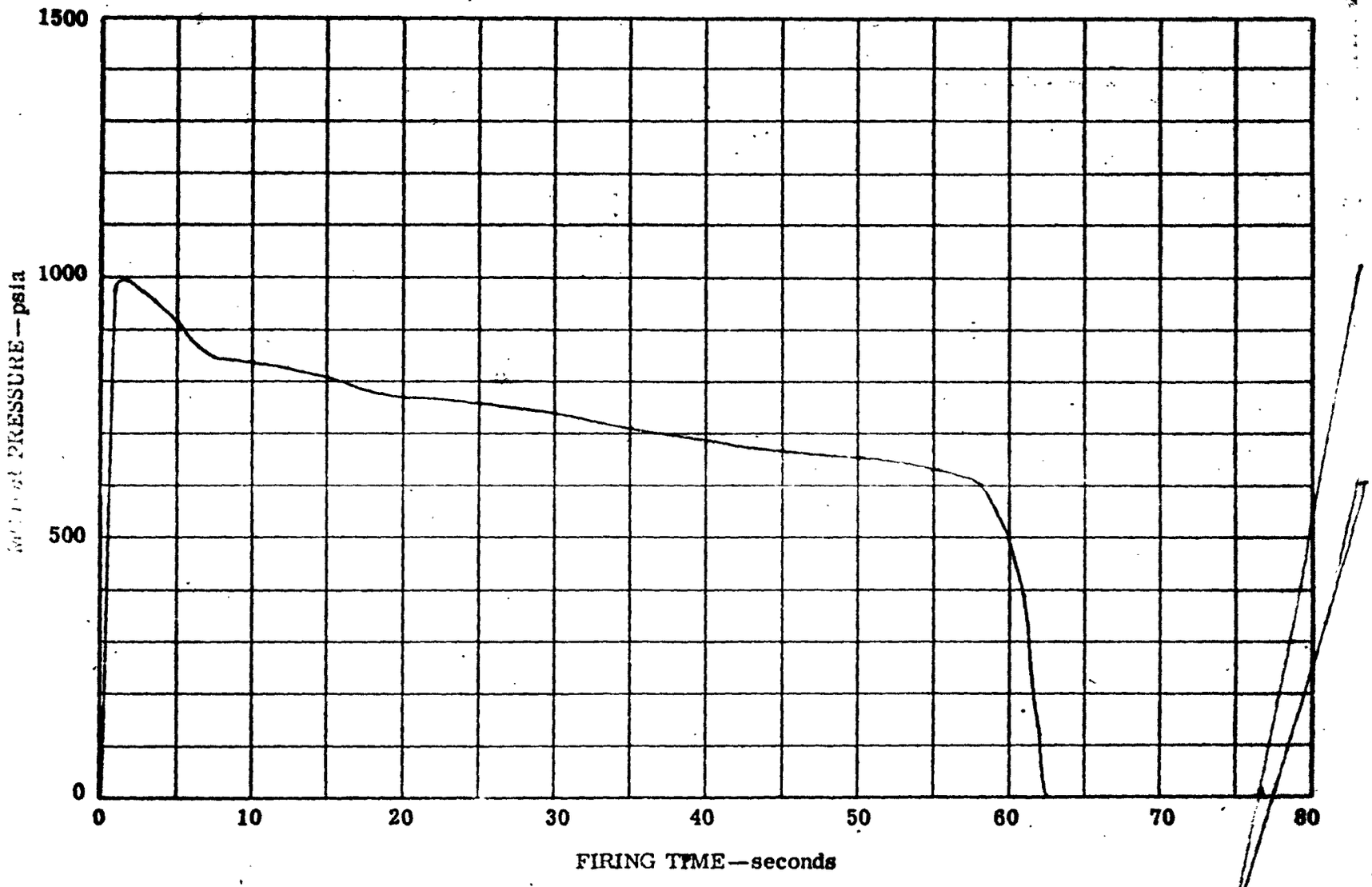



Figure 13. Motor Pressure Trace for Firing M-325

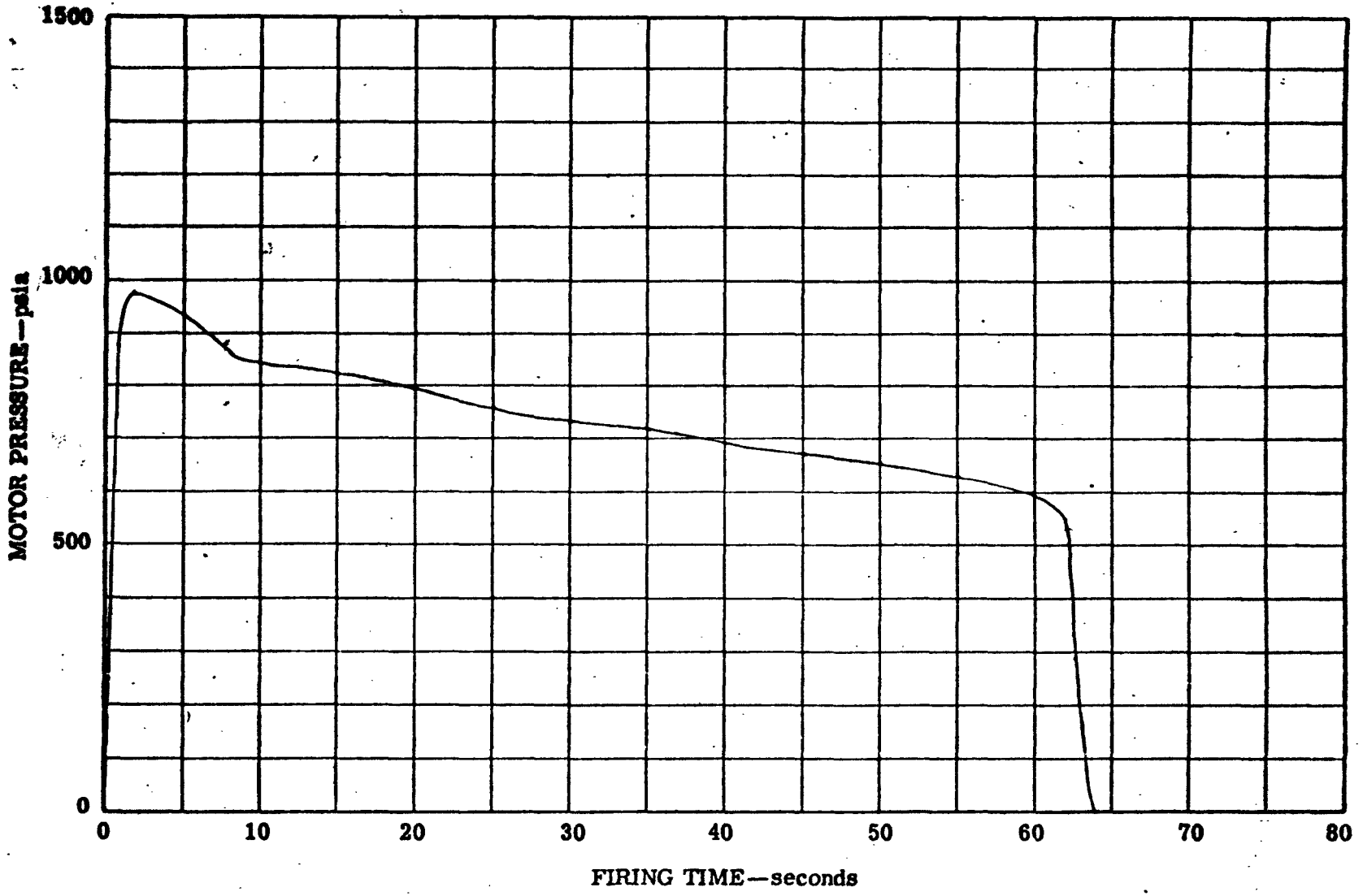



Figure 14. Motor Pressure Trace for Firing M-326

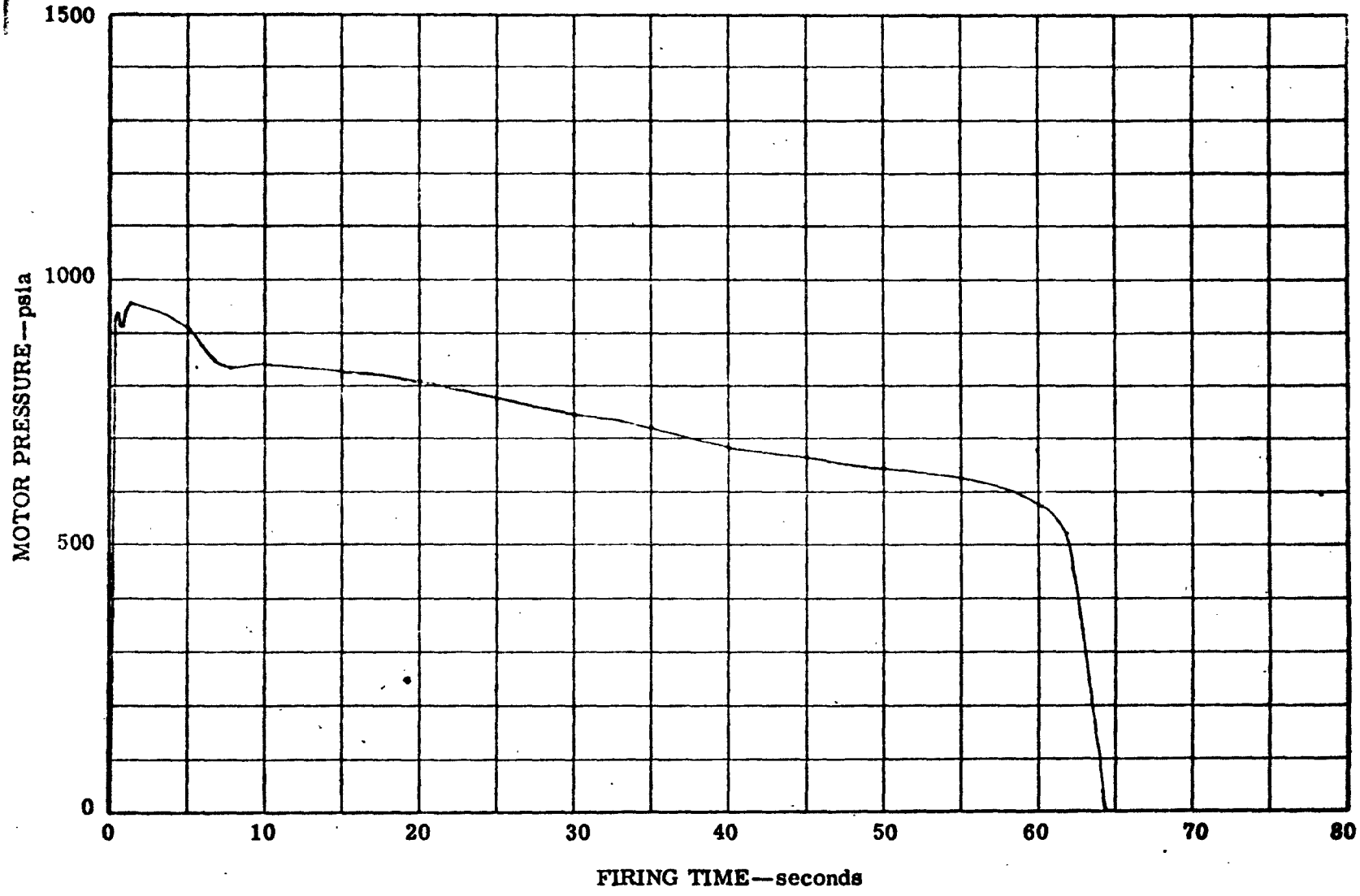



Figure 15. Motor Pressure Trace for Firing M-331

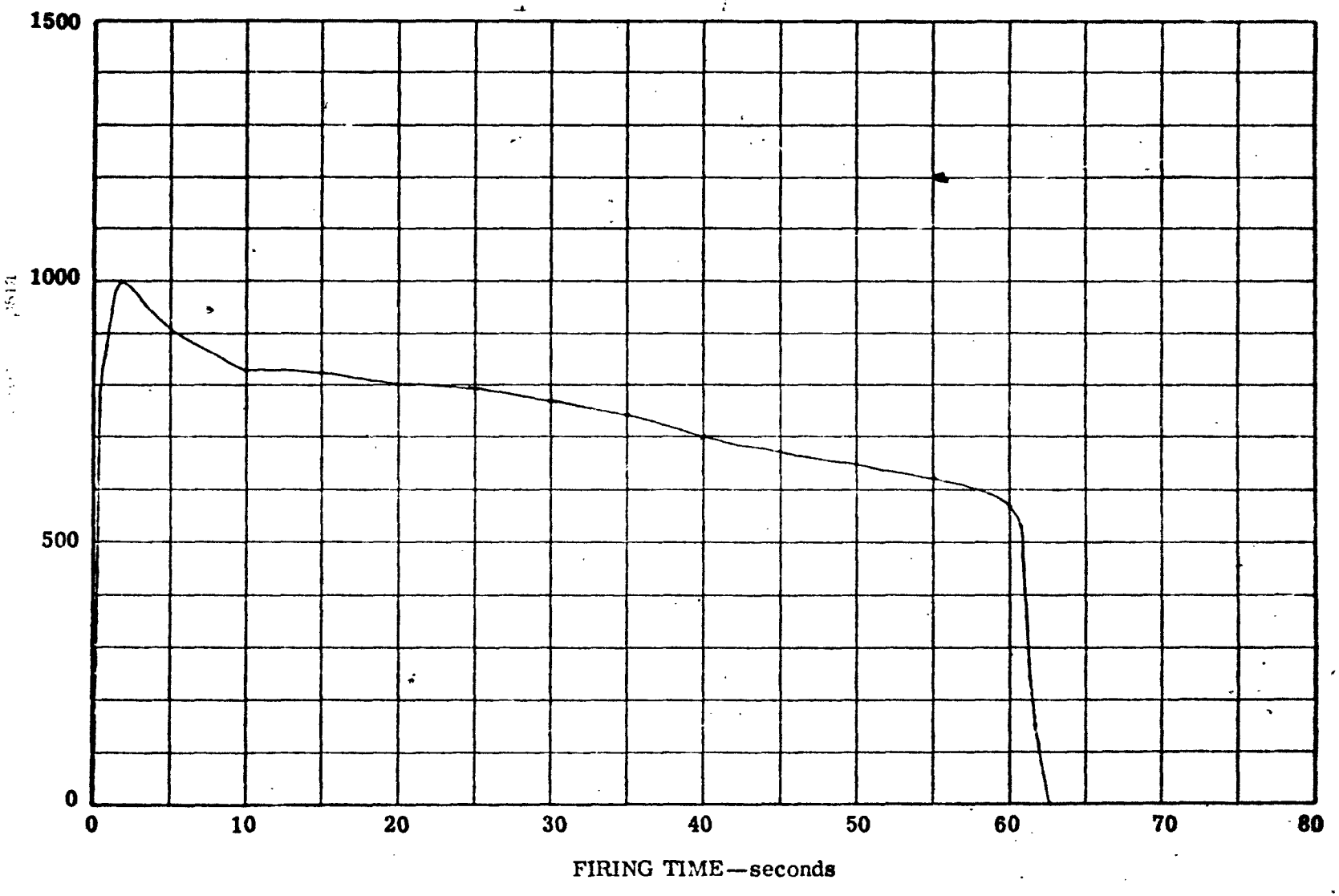



Figure 16. Motor Pressure Trace for Firing M-330

## SUMMARY AND ANALYSIS OF PROGRAM

The basic idea which generated this program was based on the concept that a ceramic powder occluded in a chromium matrix would enhance the high temperature oxidation and erosion resistance of the chromium. Electrodepositing this mixture onto graphite would then protect the graphite from the effects of hot oxidizing gases.

This "cermet" type coating initially developed under previous programs, was to be optimized during this program by a systematic study of all of the process variables.

It is believed that the work conducted on this program did result in a much greater understanding of the effect of process variables. However, most variation in the process did not appear to produce either an improved or inferior coating.

Perhaps the methods available today for evaluating these process variables or the coating itself are not sufficiently distinguishing to point out minor variations in the properties of the coating.

It is believed that the electrodeposited cermet coated in its most improved form does protect graphite from oxidation. The question that has not been answered is what are

the maximum conditions of temperature, pressure and corrosion which the coating can withstand before it fails.

## RECOMMENDATIONS FOR FURTHER RESEARCH

Studies of many fired nozzles have revealed that the coating is removed because it separates at the interface between the coating and the graphite. The adherence here is a mechanical bond and therefore subject to thermal shock.

Further research should develop the electrodeposited coating so that a gradient exists between it and the substrate.

During the final phase of the program, a few nozzles were coated with chromium-zirconium diboride cermet and then carburized in an attempt to convert the chromium to chromium carbide. Very sparse data was obtained on these nozzles due to time limitations, but the carbide conversion did appear to cause diffusion and a tighter bond.

The experience gained from this program has lead Value Engineering Company engineers to believe that the formation of carbide or other ceramic "in situ" coatings would be the next logical step in the development of protective coatings for graphite.

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| Commander<br>Air Force Flight Test Center<br>Edwards Air Force Base, California<br>Attention: FTRS | 1 |
| Commander<br>Armed Services Technical Information Agency<br>Arlington Hall Station<br>Arlington 12, Virginia<br>Attention: TIPCR | 10 |

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| Commanding General<br>Aberdeen Proving Ground<br>Aberdeen, Maryland<br>Attention: ORDBG-BLI<br>Ballistic Research Laboratories | 1 |
| Commander<br>Army Rocket and Guided Missile Agency<br>Redstone Arsenal, Alabama<br>Attention: Technical Library<br>ORDXR-OTL | 2 |
| Commanding General<br>Ordnance Ammunition Command<br>Joliet, Illinois<br>Attention: ORDLY-AREL, Engr. Library | 1 |
| Atlantic Research Corporation<br>Shirley Highway and Edsall Road<br>Alexandria, Virginia | 1 |
| Solid Propellant Information Agency<br>Applied Physics Laboratory<br>The Johns Hopkins University<br>Silver Spring, Maryland | 3 |
| Thiokol Chemical Corporation<br>Redstone Division<br>Huntsville, Alabama<br>Attention: Technical Director | 1 |
| Rocketdyne<br>6633 Canoga Avenue<br>Canoga Park, California<br>Attention: Library, Department 596-306 | 1 |
| Grand Central Rocket Company<br>P. O. Box 111<br>Redlands, California<br>Attention: Helen Ashman, Librarian | 1 |
| Aerojet-General Corporation<br>Box 1168<br>Sacramento, California<br>Attention: R. G. Weitz<br>Head, Technical Information Center | 2 |

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| Thiokol Chemical Corporation<br>Utah Division<br>Brigham City, Utah<br>Attention: Technical Director | 1 |
| Hercules Powder Company<br>Bacchus Works<br>Magna, Utah<br>Attention: Librarian | 1 |
| Lockheed Aircraft Corporation<br>Technical Information Center<br>3251 Hanover Street<br>Palo Alto, California<br>Attention: W. A. Kozumplik, Manager | 1 |
| General Electric Company<br>Cincinnati 15, Ohio<br>Attention: Technical Information Center | 1 |
| Defense Metals Information Center<br>Battelle Memorial Institute<br>505 King Avenue<br>Columbus 1, Ohio | 1 |
| Clevite Corporation<br>Mechanical Research Division<br>540 East 105th Street<br>Cleveland 8, Ohio<br>Attention: Mr. Gail Davies | 1 |
| United Technology Corporation<br>P. O. Box 358<br>Sunnyvale, California<br>Attention: Librarian | 1 |
| Edward Sheehy<br>Allegany Ballistics Laboratory<br>Cumberland, Maryland | 1 |
| Earl T. Hayes<br>The Pentagon<br>Room 3D1085<br>Washington 25, D. C. | 1 |
| Milton Levy<br>U.S. Army Ordnance<br>Watertown Arsenal<br>Watertown 72, Massachusetts | 1 |

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